**DERWENT-ACC-NO:** 2003-602746

**DERWENT-WEEK:** 200357



**TITLE:** Slurry regeneration method for mechanical processing slurry, involves concentration to remove dispersion medium, pH adjustment and recovery of ground particles

Matsuo et. al.

**PATENT-ASSIGNEE:** AGURU JAPAN KK[AGURN]

**PRIORITY-DATA:** 2001JP-0397388 (December 27, 2001)

**PATENT-FAMILY:**

| PUB-NO | PUB-DATE | LANGUAGE | PAGES | MAIN-IPC |
|---|---|---|---|---|
| JP **2003200347** A | July 15, 2003 | N/A | 007 | B24B 057/02 |

**APPLICATION-DATA:**

| PUB-NO | APPL-DESCRIPTOR | APPL-NO | APPL-DATE |
|---|---|---|---|
| JP2003200347A | N/A | 2001JP-0397388 | December 27, 2001 |

**INT-CL (IPC):** B01D035/02, B24B057/02 , C02F001/44 , C02F011/12 , H01L021/304

**ABSTRACTED-PUB-NO:** JP2003200347A

**BASIC-ABSTRACT:**

NOVELTY - A slurry comprising ground particles, dispersion medium (DM) and impurities, is concentrated to remove DM which is collected separately. pH of the concentrated slurry is adjusted and the ground particles of particle size less than a predetermined value are recovered.

DETAILED DESCRIPTION - An INDEPENDENT CLAIM is also included for slurry regenerating apparatus.

USE - To recycle and regenerate slurry.

ADVANTAGE - Amount of slurry discarded after regeneration is highly minimized, which improves the recycling rate of slurry.

DESCRIPTION OF DRAWING(S) - The figure shows the general view of a slurry regenerating apparatus.

filter for concentration 1

filter for recovery 5

pH controller 17

recovery unit 20

**CHOSEN-DRAWING:** Dwg.4/7

**TITLE-TERMS:** SLURRY REGENERATE METHOD MECHANICAL PROCESS SLURRY CONCENTRATE REMOVE DISPERSE MEDIUM PH ADJUST RECOVER GROUND PARTICLE

**DERWENT-CLASS:** D15 P61 U11

**CPI-CODES:** D04-A;

**EPI-CODES:** U11-C06A1A; U11-C15Q;

**SECONDARY-ACC-NO:**

**CPI Secondary Accession Numbers:** C2003-164406

**Non-CPI Secondary Accession Numbers:** N2003-480325

**PAT-NO:** JP02003200347A

**DOCUMENT-IDENTIFIER:** JP **2003200347** A

**TITLE:** SLURRY RECYCLING METHOD AND SLURRY RECYCLING DEVICE USING THE SAME

**PUBN-DATE:** July 15, 2003

**INVENTOR-INFORMATION:**

| NAME | COUNTRY |
|---|---|
| MATSUO, TAKASHI | N/A |
| MIZUTA, TAKASHI | N/A |
| AOTO, KOJI | N/A |

**ASSIGNEE-INFORMATION:**

| NAME | COUNTRY |
|---|---|
| AGRU JAPAN:KK | N/A |

**APPL-NO:** JP2001397388

**APPL-DATE:** December 27, 2001

**INT-CL (IPC):** B24B057/02 , B01D035/02 , C02F001/44 , C02F011/12 , H01L021/304

**ABSTRACT:**

PROBLEM TO BE SOLVED: To solve such a problem that used slurry discharged from CMP or the like, which contains impurities such as pad chips, metal chips and oxide film chips and is diluted with a cleaning agent and the like, is difficult to reuse as it is and is disposed of as waste.

SOLUTION: A slurry recycling device, which is for slurry with abrasive grains, a dispersion medium and impurities, comprises a circulation part having a concentration part for removing part of the dispersion medium from the slurry and a pH regulation part for regulating the pH value of the slurry, and a recovery part connected to the circulation part to recover the dispersion medium and abrasive grains of a given grain size or smaller from the slurry.

L23 ANSWER 8 OF 39 CAPLUS COPYRIGHT 2007 ACS on STN
Full Text
AN 2003:541542 CAPLUS
DN 139:103071
ED Entered STN: 16 Jul 2003
TI Method and apparatus for **regeneration** of spent **polishing slurry**
IN Matsuo, Takashi; Mizuta, Takashi; Aoto, Koji
PA Agru Japan Co., Ltd., Japan
SO Jpn. Kokai Tokkyo Koho, 7 pp.
CODEN: JKXXAF
DT Patent
LA Japanese
IC ICM B24B057-02
ICS B01D035-02; C02F001-44; C02F011-12; H01L021-304
CC 48-1 (Unit Operations and Processes)
Section cross-reference(s): 60, 76
FAN.CNT 1

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|---|---|---|---|---|
| PI JP 2003200347 | A | 20030715 | JP 2001-397388 | 20011227 |
| PRAI JP 2001-397388 | | 20011227 | | |

CLASS

| PATENT NO. | CLASS | PATENT FAMILY CLASSIFICATION CODES |
|---|---|---|
| JP 2003200347 | ICM | B24B057-02 |
| | ICS | B01D035-02; C02F001-44; C02F011-12; H01L021-304 |
| | IPCI | B24B0057-02 [ICM,7]; B24B0057-00 [ICM,7,C*]; B01D0035-02 [ICS,7]; B01D0035-00 [ICS,7,C*]; C02F0001-44 [ICS,7]; C02F0011-12 [ICS,7]; H01L0021-304 [ICS,7]; H01L0021-02 [ICS,7,C*] |
| | IPCR | B24B0057-00 [I,C*]; B24B0057-02 [I,A]; B01D0035-00 [I,C*]; B01D0035-02 [I,A]; C02F0001-44 [I,C*]; C02F0001-44 [I,A]; C02F0011-12 [I,C*]; C02F0011-12 [I,A]; H01L0021-02 [I,C*]; H01L0021-304 [I,A] |

AB In **regeneration** of spent **polishing slurry** contg. **abrasive** grains, dispersing medium and impurities, the following processes are included: a **slurry** concn. process for removing a part of the dispersing medium from the **slurry**, a **pH** adjustment process for adjusting **pH** of the concd. **slurry**, and a recovery process for recovering **abrasive** grains of less than or equal to a prescribed particle diam. and dispersing medium from the **pH** adjusted **slurry**. The spent **polishing slurry** is discharged from chem.-mech. **polishing** process, etc.

ST spent **polishing slurry regeneration** app; chem mechanic **polishing** spent **slurry regeneration** app

IT **pH**
(adjustment of; method and app. for **regeneration** of spent **polishing slurry** from **polishing** process)

IT **Polishing**
(chem.-mech., spent **slurry** from; method and app. for **regeneration** of spent **polishing slurry** from **polishing** process)

IT **Abrasives**
(grains, recovery of; method and app. for **regeneration** of spent **polishing slurry** from **polishing** process)

IT Dispersion (of materials)
(medium for, recovery of; method and app. for **regeneration** of spent **polishing slurry** from **polishing** process)

IT Concentration (process)

(19)日本国特許庁（ＪＰ）　(12)**公開特許公報（Ａ）**　(11)特許出願公開番号
特開2003－200347
（P2003－200347A）
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| (51)Int.Cl.⁷ | 識別記号 | ＦＩ | テーマコード（参考） |
|---|---|---|---|
| Ｂ２４Ｂ　57/02 | | Ｂ２４Ｂ　57/02 | ３Ｃ０４７ |
| Ｂ０１Ｄ　35/02 | | Ｃ０２Ｆ　1/44 | Ｆ　４Ｄ００６ |
| Ｃ０２Ｆ　1/44 | | | ＺＡＢＥ　４Ｄ０５９ |
| | ＺＡＢ | 11/12 | Ｚ　４Ｄ０６４ |
| 11/12 | | Ｈ０１Ｌ　21/304 | ６２２Ｅ |

審査請求　有　請求項の数10　ＯＬ　（全７頁）　最終頁に続く

(21)出願番号　特願2001－397388(P2001－397388)

(22)出願日　平成13年12月27日(2001.12.27)

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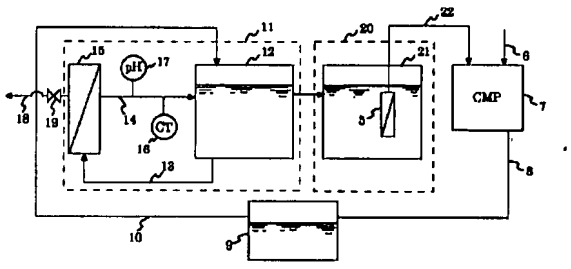
(54)【発明の名称】　スラリ再生方法およびそれを用いたスラリ再生装置

(57)【要約】
【課題】　ＣＭＰ等から排出される使用済みスラリは、パッド片、金属片、酸化膜片等の不純物を含有するとともに、洗浄剤等により希釈されているため、そのままの状態で再利用するのは困難であり廃棄処分されていた。
【解決手段】　砥粒、分散媒および不純物を有するスラリの再生装置において、前記スラリより分散媒の一部を除去する濃縮部と前記スラリのｐＨ値を調整するｐＨ調整部とを有する循環部と、前記循環部と接続され、前記スラリより分散媒および所定粒径以下の砥粒を回収する回収部とを具備することを特徴とするスラリ再生装置。



1

【特許請求の範囲】

【請求項１】　砥粒、分散媒および不純物を有するスラリの再生方法において、

前記スラリより前記分散媒の一部を除去するスラリ濃縮工程と、

前記濃縮されたスラリのｐＨ値を調整するｐＨ調整工程と、

前記ｐＨ値が調整されたスラリより所定粒径以下の砥粒および分散媒を回収する回収工程とを具備することを特徴とするスラリ再生方法。

【請求項２】　砥粒、分散媒および不純物を有するスラリの再生方法において、

前記スラリのｐＨ値を調整するｐＨ調整工程と、

前記ｐＨ値が調整されたスラリより前記分散媒の一部を除去するスラリ濃縮工程と、

前記濃縮されたスラリより所定粒径以下の砥粒および分散媒を回収する回収工程とを具備することを特徴とするスラリ再生方法。

【請求項３】　砥粒、分散媒および不純物を有するスラリの再生方法において、

前記スラリより前記分散媒の一部を除去しながら、ｐＨ値を調整する工程と、

前記濃縮およびｐＨ調整がされたスラリより所定粒径以下の砥粒および分散媒を回収する回収工程とを具備することを特徴とするスラリ再生方法。

【請求項４】　前記濃縮工程は、前記スラリを分散媒のみを通過させる濃縮用フィルタに接触させ、この濃縮用フィルタを通過した分散媒を除去することにより行うことを特徴とする請求項１乃至３のいずれか１項記載のスラリ再生方法。

【請求項５】　前記ｐＨ値を特定の範囲内に調整することを特徴とする請求項１乃至４のいずれか１項記載のスラリ再生方法。

【請求項６】　前記ｐＨ値の調整において、

前記スラリに分散剤を添加することによりｐＨ値を調整することを特徴とする請求項１乃至５のいずれか１項記載のスラリ再生方法。

【請求項７】　前記回収工程は、前記スラリを分散媒および所定粒径以下の砥粒を通過させる回収用フィルタに通過させ、この回収用フィルタを通過した成分を回収することにより行うことを特徴とする請求項１乃至６のいずれか１項記載のスラリ再生方法。

【請求項８】　砥粒、分散媒および不純物を有するスラリの再生装置において、

前記スラリより分散媒の一部を除去する濃縮部と、前記スラリのｐＨ値を調整するｐＨ調整部とを有する循環部と、

前記循環部と接続され、前記スラリより分散媒および所定粒径以下の砥粒を回収する回収部とを具備することを特徴とするスラリ再生装置。

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【請求項９】　前記濃縮部は、分散媒だけを通過させる濃縮用フィルタを具備することを特徴とする請求項８記載のスラリ再生装置。

【請求項１０】　前記スラリ回収部は、分散媒および所定粒径以下の砥粒を通過させる回収用フィルタを具備することを特徴とする請求項８または９記載のスラリ再生装置。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、使用済みスラリを再生するスラリ再生方法およびそれを利用したスラリ再生装置に関し、特に廃棄するスラリの量を低減し、スラリの再生率を向上させたスラリ再生方法およびそれを用いたスラリ再生装置に関する。

【０００２】

【従来の技術】現在、半導体生産においてはＣＭＰ（Ｃｈｅｍｉｃａｌ　Ｍｅｃｈａｎｉｃａｌ　Ｐｏｌｉｓｈｉｎｇ：化学的機械的研磨）が必須の技術となっている。このＣＭＰは、酸化膜をはじめｍｅｔａｌ配線にも適用されるようになっており、さらにその適用範囲が拡大する傾向にある。

【０００３】ＣＭＰ工程では砥粒を分散させたスラリを用いてデバイス表面を平坦化加工するものであり、多量の洗浄水および砥粒を必要とする。現在、このような多量の洗浄水および砥粒は廃液として処分されており、環境への負荷の低減、資源の有効利用の観点からこれらの再利用が望まれている。また、ＣＭＰ工程に用いられる砥粒としては、通常、$SiO_2$等からなる市販の砥粒が用いられているが、これらは非常に高価であり、経済的な観点からも砥粒の使用量を少なくすることが望まれている。

【０００４】

【発明が解決しようとする課題】上記したようにＣＭＰ工程に用いられる洗浄水、砥粒等は資源の有効利用、環境への配慮、経済的な観点から廃棄量を低減し再利用することが望ましい。特に、ＣＭＰ工程に用いられる砥粒は非常に高価であり、その使用量を極力減少させることが望ましい。

【０００５】しかしながら、一度使用したスラリは洗浄水等により希釈されているため、スラリ濃度が低下し、ｐＨ値も中性側に変化している。スラリ濃度が低下すると、所望の研磨が行えなくなる可能性がある。また、スラリのｐＨ値が中性側に変化すると、砥粒の凝集が発生し、所望の研磨が行えなくなる可能性がある。

【０００６】さらに、一度使用したスラリには、研磨屑、例えばパッド片、金属片、酸化膜片等が混入している。上記したようなスラリを用いてＣＭＰ工程を行った場合、適切な研磨を行うことができなくなる。特に、近年の高集積化された半導体デバイスの加工においては精密な加工が求められており、この点からもスラリの特性

を一定に保つことが重要である。

【0007】本発明は、上記したような課題を解決するためになされたものであって、廃棄するスラリの量を低減しスラリの再生率を向上させるとともに、新規なスラリと同等の特性を有するスラリを得るためのスラリ再生方法およびそれを用いたスラリ再生装置を提供することを目的としている。

【0008】

【課題を解決するための手段】本発明のスラリ再生方法は、砥粒、分散媒および不純物（研磨屑、パッド片、金属片、酸化膜片等）を有するスラリの再生方法において、前記スラリより前記分散媒の一部を除去するスラリ濃縮工程と、前記濃縮されたスラリのｐＨ値を調整するｐＨ調整工程と、前記ｐＨ値が調整されたスラリより所定粒径以下の砥粒および分散媒を回収する回収工程とを具備することを特徴とする。

【0009】また、本発明のスラリ再生方法は、砥粒、分散媒および不純物を有するスラリの再生方法において、前記スラリのｐＨ値を調整するｐＨ調整工程と、前記ｐＨ値が調整されたスラリより前記分散媒の一部を除去するスラリ濃縮工程と、前記濃縮されたスラリより所定粒径以下の砥粒および分散媒を回収する回収工程とを具備することを特徴とする。

【0010】本発明のスラリ再生方法では、上記したようにスラリ濃縮工程とｐＨ調整工程とを別々に行う以外に、これらを同時に行ってもよい。

【0011】前記濃縮工程は、例えば前記スラリを分散媒のみを通過させる濃縮用フィルタに接触させ、この濃縮用フィルタを通過した分散媒を除去することにより行うことができる。

【0012】前記ｐＨ値は、特定の範囲内に調整することが好ましく、例えば酸化膜用スラリでは１０～１１の範囲内である。このようなｐＨ値の調整は、例えば水酸化カリウム等の分散剤を添加することにより行うことができる。

【0013】前記回収工程は、例えば前記スラリを分散媒および所定粒径以下の砥粒を通過させる回収用フィルタに通過させ、この回収用フィルタを通過した成分を回収することにより行うことができる。

【0014】また、本発明のスラリ再生装置は、砥粒、分散媒および不純物を有するスラリの再生装置において、前記スラリより分散媒の一部を除去する濃縮部と、前記スラリのｐＨ値を調整するｐＨ調整部とを有する循環部と、前記循環部と接続され、前記スラリより分散媒および所定粒径以下の砥粒を回収する回収部とを具備することを特徴とする。

【0015】前記濃縮部は、例えば分散媒だけを通過させる濃縮用フィルタを具備し、前記スラリ回収部は、例えば分散媒および所定粒径以下の砥粒を通過させる回収用フィルタを具備するものである。

【0016】

【発明の実施の形態】本発明の実施の形態について説明する。

【0017】図1は、本発明のスラリ再生方法の例を示したものである。図1（ａ）に示されるスラリ再生方法は、まず最初に使用済みスラリより不要な分散媒（水分、分散剤、洗浄液等からなる液体成分）を除去してスラリを濃縮し、この濃縮されたスラリに分散剤を加えてｐＨを調整し、最後に必要とされる砥粒および分散媒を回収するものである。図1（ｂ）に示されるスラリ再生方法は、まず最初に使用済みスラリに分散剤を加えてｐＨを調整し、このｐＨが調整されたスラリより不要な分散媒を除去してスラリを濃縮し、最後に必要とされる砥粒および分散媒を回収するものである。

【0018】このような濃縮とｐＨ調整とは必ずしも別々に行う必要はなく、例えば図1（ｃ）に示されるようにｐＨ調整を行いながら、濃縮を行っても構わない。このように、必要とされる砥粒および分散媒を回収する工程の前にｐＨを調整することで、砥粒の効率的な回収が可能となる。

【0019】以下、本発明のスラリ再生方法について、図1（ａ）を例として説明する。図2は濃縮工程における濃縮方法を示した一例を示したものである。なお図2において、図面右側は濃縮側であり、濃縮されるスラリを示している。図面左側は排出側であり、スラリより除去される分散媒を示している。本発明では濃縮用フィルタ1として、例えば主として分散媒2のみを通過させ、砥粒3や不純物4を通過させないものを用いる。砥粒3としては、例えば酸化膜用としては酸化セリウム（$CeO_2$）、シリカ（$SiO_2$）等が挙げられ、W、Alおよび Cu用としてはアルミナ（$Al_2O_3$）等が挙げられる。濃縮用フィルタとしては、用いる砥粒によっても異なるが、例えば孔径が０．０１～０．１５μｍである中空糸膜が挙げられる。特に、酸化膜用としては孔径が０．１μｍ程度、W用としては０．０５μｍ程度のものが好ましい。

【0020】このような濃縮用フィルタ１の濃縮側より濃縮させようとするスラリ、例えば使用済みスラリを接触させ、分散媒２の一部を濃縮用フィルタ１を通過させて除去する。この際、濃縮用フィルタ１を通過できない砥粒３や不純物４は濃縮側のスラリに残留する。この濃縮工程により主として分散媒のみを分離除去することができるため、濃縮側のスラリの濃度を上げることができる。なお、この濃縮工程では全ての分散媒を除去する必要はなく、所望とするスラリ濃度に対して過剰な分の分散媒を除去すればよい。

【0021】また、本発明におけるｐＨ調整工程はスラリに分散剤を添加して、スラリのｐＨ値を調整するものである。使用済みのスラリには洗浄液等が混入しているため、新規なスラリよりもｐＨ値が中性側に変化してい

る。このようにｐＨ値が変化した場合、砥粒等が凝集してしまう。このような場合、後に説明する回収工程における回収率が低くなり、また、このようなスラリをＣＭＰ装置等で再使用した場合、研磨の均一性等が新規なスラリと異なってしまうことがある。本発明ではスラリのｐＨ値を調整することにより、上記したような課題を解消することができる。

【００２２】このようなｐＨ調整を行うための分散剤としては、例えば酸化膜用スラリにはＫＯＨ（水酸化カリウム）、$NH_4OH$（水酸化アンモニウム）等、Ｃｕ用スラリには有機酸等、ＷおよびＡｌ用スラリには$H_2O_2$（過酸化水素）等を添加することにより行うことができる。スラリのｐＨ値は、例えば酸化膜用スラリでは１０～１１の範囲内、Ｃｕ、ＷおよびＡｌ用スラリでは２～３の範囲内とすることで、砥粒の凝集を解消し、新規なスラリとほぼ同等の研磨特性を得ることが可能となる。

【００２３】また、図３は回収工程における回収方法を示したものである。図面左側は被回収側であり、回収されるべき成分を含むもの、例えば上記濃縮やｐＨ調整等が行われたスラリが配置される。また、図面右側は回収側であり、被回収側より回収された分散媒２と砥粒３が位置する。

【００２４】本発明における回収工程では、回収用フィルタ５として、一定以上の大きさの不純物を通過させず、所望とする分散媒および砥粒のみを通過させるような回収用フィルタ５を用いる。このような回収用フィルタとしては、用いる砥粒によっても異なるが、例えば孔径が０．１～３μｍであるメンブレンフィルタが挙げられる。特に、酸化膜用スラリの場合には孔径が１μｍ程度のものが好ましい。

【００２５】このような回収用フィルタ５の被回収側に濃縮やｐＨ調整が行われたスラリを配置すると、使用済みスラリに含まれる砥粒３や分散媒２は回収用フィルタ５を通過できるため回収側へ移動する。また、不純物４のように大きなものは回収用フィルタ５を通過できないため、被回収側に残留する。

【００２６】このように回収用フィルタ５を通過したものだけを回収することで、不純物が含まれないスラリを得ることができる。また、このような回収用フィルタ５を用いると分散媒２と砥粒３とを同等の割合で通過させることができるため、被回収側の濃度を適切に調整した後に回収工程を行うことで、再度濃度の調整を行う必要がない。特に、スラリのｐＨ値を適切に調整し、砥粒の凝集を解消した後に被回収側に配置することで、効率的な砥粒の回収が可能となる。このようにして回収されたスラリは再びＣＭＰ装置で使用することができる。

【００２７】以上説明したように、本発明ではスラリより主として分散媒を除去してスラリの濃度を調整するとともに、スラリより必要な成分のみを回収し、不必要な成分を回収しないことで、廃棄する成分を極力少なくし、スラリの再生効率を向上させることが可能となる。また、回収工程の前にスラリのｐＨ調整を行うことで、スラリの効率的な回収も可能となる。

【００２８】次に本発明のスラリ再生装置について説明する。

【００２９】図４は、本発明のスラリ再生装置の一例を示した概略図である。新規なスラリはスラリ導入管６よりＣＭＰ装置７へ送られる。このＣＭＰ装置７で使用されることにより、新規なスラリには洗浄液、パッド屑等が混入し、使用済みスラリとなる。使用済みスラリは使用済みスラリ排出管８を通して貯蔵タンク９へ一端貯蔵される。そして、貯蔵タンク９より配管１０を通して循環部１１へと送られる。

【００３０】循環部１１は循環用タンク１２とこれに接続された循環用配管１３、１４とからなり、この循環路の途中に濃縮部１５、濃度測定部１６およびｐＨ調整部１７が配置される。

【００３１】循環用タンク１２に蓄えられた使用済みスラリは循環用配管１３を通して濃縮部１５に送られる。この濃縮部１５には例えば図１に示されるような分散媒のみを通過させて除去する濃縮用フィルタ（分散媒除去フィルタ）１が設けられている。濃縮部１５に送られたスラリのうち、分散媒の一部が濃縮用フィルタ１を通過し、分散媒排出管１８を通して外部へ排出される。また、濃縮用フィルタ１を通過しなかった砥粒、不純物および分散媒は、濃縮部１５に接続された循環用配管１４を通して循環用タンク１２に戻される。

【００３２】このようにして、スラリを濃縮部１５を介して循環させることで、スラリより過剰な分散媒を除去して、スラリの濃度を上げることができる。このスラリの濃度は、例えば循環路の途中に設けられた濃度測定部１６により監視して、所定の濃度に達したら分散媒排出用配管１８に設けられた弁１９を閉じて分散媒の排出を停止して、濃度の上昇を抑制することができる。

【００３３】次に、弁１９を閉じた状態でスラリを再度循環させながらｐＨ調整部１７より分散剤を添加して、ｐＨ値を所定の値に調整する。ｐＨ値を調整することにより、砥粒の凝集を防止し、次の回収工程で効率的な回収を可能とすることができる。

【００３４】このｐＨ調整は濃縮の前に行ってもよく、また濃縮と同時に行ってもよい。濃縮とｐＨ調整とを同時に行う場合には、分散媒排出用配管１８に設けられた弁１９を開いた状態でスラリを循環させて、分散媒を除去するとともに、ｐＨ調整部１７より分散剤を添加してｐＨ調整を行う。このようにすることで、ｐＨ調整と濃縮とを同時に行うことができ、スラリの再生時間を大幅に短縮することができる。

【００３５】次に、濃縮およびｐＨ調整が行われたスラリは、回収部２０へと送られる。回収部２０は主として

回収用タンク２１および回収用フィルタ５とからなる。回収用フィルタ５は、例えば図３に示されるように、不純物を通過させず、分散媒および砥粒のみを通過させるようなものである。このようなフィルタとしては、中空糸膜等を用いることができる。

【００３６】この回収用フィルタ５は、その被回収側が回収用タンク２１に蓄えられたスラリと接触するようにし、回収側が回収用配管２２へと接続されている。回収用タンク２１に蓄えられたスラリのうち、分散媒および一定粒径以下の砥粒は回収用フィルタ５を通過し、回収用配管２２を通してＣＭＰ装置７へ送られ、再使用される。また、回収用フィルタ５を通過できない不純物は、そのまま回収用タンク２１に蓄積される。

【００３７】

【実施例】以下、本発明の実施の形態について実施例を参照して説明する。

【００３８】スラリ再生装置として図４に示されるような構成の装置を作製した。ＣＭＰ装置に主として$SiO_2$（粒径　約０．０５～１μｍ）からなり、分散剤としてＫＯＨ（水酸化カリウム）を含む新規なスラリ（キャボット・コーポレーション製、ＳＣ－１）を導入し、このＣＭＰ装置から排出される使用済みスラリを得た。この使用済みスラリを循環部に導入し、濃縮と同時に分散剤であるＫＯＨを添加してｐＨ調整を行った。なお、濃縮用フィルタとしては、ＭＳ－８１０２（孔径０．１μｍ）を用い、スラリのｐＨ値は１１に調整した。

【００３９】次に、濃縮およびｐＨ調整が行われたスラリを回収用タンクへ導入し、回収用フィルタを使用してスラリより所望とされる分散媒および砥粒を回収し再生スラリとした。なお、回収用フィルタとしては、ＣＭＰ４１０－１０ＵＶ－Ｍ３（孔径１μｍ）を用いた。

【００４０】このようにして得られた再生スラリの粒度分布を図５に示す。図５に示されるように、本発明のスラリ再生装置により得られた再生スラリには、使用済みスラリに見られたような３μｍ～８μｍ付近の不純物がなく、かつ粒度分布も新規スラリとほぼ同等のものとなった。

【００４１】さらに、このような再生スラリを使用して実際にウェハを研磨し、その研磨レート（Å／ｍｉｎ）および研磨されたウェハの均一性（ウェハの凹凸）を測定した。結果を新規スラリを使用した場合と合わせて図６、図７に示す。

【００４２】図６、図７に示されるように、いずれの試料においても再生スラリを使用した場合と新規スラリを使用した場合とで大差はなく、本発明の再生スラリは新規スラリとほぼ同等の特性を有することが認められた。

【００４３】

【発明の効果】本発明のスラリ再生方法、再生装置によれば、使用済みスラリより所望とする粒径以下の砥粒を回収する工程の前に、濃縮およびｐＨ調整を行うことで、使用済みスラリの効率的な再生を実現することが可能となる。

【図面の簡単な説明】

【図１】本発明のスラリ再生方法の工程を示した図。

【図２】本発明における濃縮方法の一例を示した概観図。

【図３】本発明における回収方法の一例を示した概観図。

【図４】本発明のスラリ再生装置を示した概観図。

【図５】再生スラリの粒度分布を示した図。

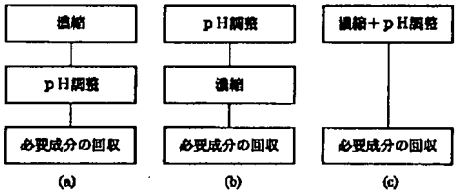
【図６】再生スラリを使用した研磨における研磨レートの結果を示した図。

【図７】再生スラリを使用した研磨におけるウェハの均一性を示した図。

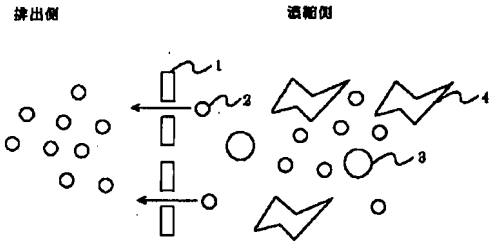
【符号の説明】

１……濃縮用フィルタ
５……回収用フィルタ
１１……循環部
１５……濃縮部
１７……ｐＨ調整部
２０……回収部

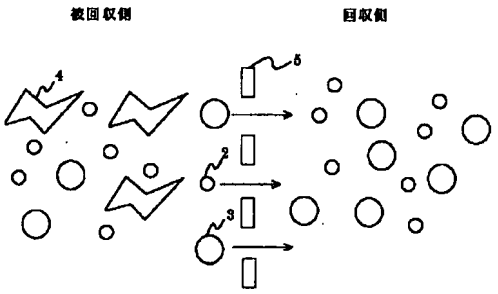
【図１】



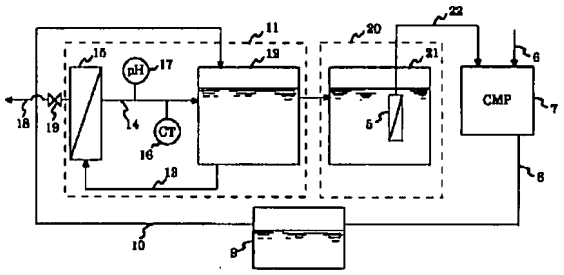

【図２】



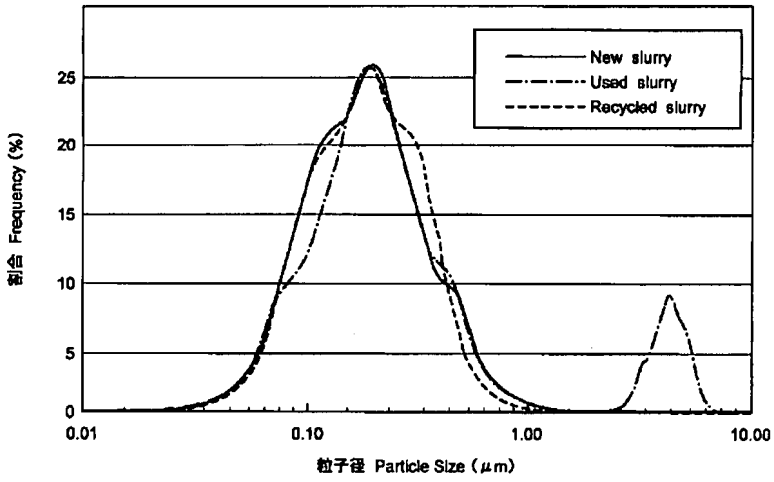
【図3】



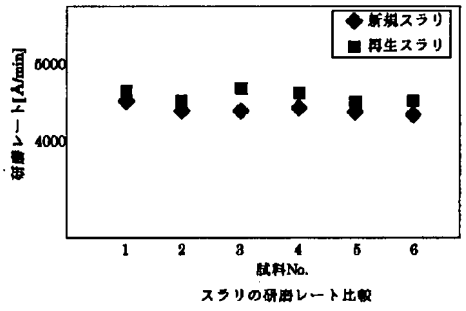

【図4】



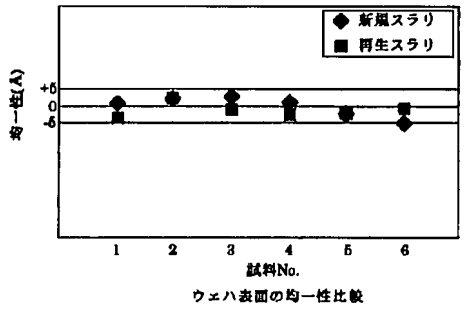

【図5】




【図6】




【図7】

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JP 2003-200347

CLAIMS

[Claim(s)]
[Claim 1] The slurry playback approach characterized by providing the slurry concentration process of removing said a part of dispersion medium from said slurry, pH adjustment process of adjusting said condensed pH value of a slurry, and the recovery process that collects the abrasive grains and dispersion media below predetermined particle size from the slurry to which said pH value was adjusted in the playback approach of the slurry which has an abrasive grain, a dispersion medium, and an impurity.
[Claim 2] The slurry playback approach characterized by providing pH adjustment process of adjusting the pH value of said slurry, the slurry concentration process of removing said a part of dispersion medium from the slurry to which said pH value was adjusted, and the recovery process that collects the abrasive grains and dispersion media below predetermined particle size from said condensed slurry in the playback approach of the slurry which has an abrasive grain, a dispersion medium, and an impurity.
[Claim 3] The slurry playback approach characterized by providing the process which adjusts a pH value, and the recovery process which collects the abrasive grains and dispersion media below predetermined particle size from the slurry to which said concentration and pH adjustment were carried out in the playback approach of the slurry which has an abrasive grain, a dispersion medium, and an impurity while removing said a part of dispersion medium from said slurry.
[Claim 4] Said concentration process is claim 1 characterized by carrying out by removing the dispersion medium which said slurry was contacted in the filter for concentration which passes only a dispersion medium, and passed this filter for concentration thru/or the slurry playback approach of three given in any 1 term.
[Claim 5] Claim 1 characterized by adjusting said pH value within the limits of specification thru/or the slurry playback approach of four given in any 1 term.
[Claim 6] Claim 1 characterized by adjusting a pH value by adding a dispersant to said slurry in adjustment of said pH value thru/or the slurry playback approach of five given in any 1 term.
[Claim 7] Said recovery process is claim 1 characterized by carrying out by collecting the components which the filter for recovery which passes the abrasive grain below a dispersion medium and predetermined particle size was made to pass said slurry, and passed this filter for recovery thru/or the slurry playback approach of six given in any 1 term.
[Claim 8] The slurry regenerative apparatus characterized by providing the circulation section which has the enriching section which removes a part of dispersion medium from said slurry, and pH controller which adjusts the pH value of said slurry in the regenerative apparatus of the slurry which has an abrasive grain, a dispersion medium, and an impurity, and the stripping section which is connected with said circulation section and collects the abrasive grains below a dispersion medium and predetermined particle size from said slurry.
[Claim 9] Said enriching section is a slurry regenerative apparatus according to claim 8 characterized by providing the filter for concentration which passes only a dispersion medium.
[Claim 10] Said slurry stripping section is a slurry regenerative apparatus according to claim 8 or 9 characterized by providing the filter for recovery which passes the abrasive grain below a dispersion

medium and predetermined particle size.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention reduces the amount of the slurry discarded especially about the slurry regenerative apparatus using the slurry playback approach and it which reproduce a used slurry, and relates to the slurry regenerative apparatus using the slurry playback approach and it which raised the regeneration rate of a slurry.
[0002]
[Description of the Prior Art] In current and semiconductor production, CMP (Chemical Mechanical Polishing: chemical mechanical polish) serves as an indispensable technique. This CMP is in the inclination which it is applied also to metal wiring including an oxide film, and that applicability expands further.
[0003] At a CMP process, flattening processing of the device front face is carried out using the slurry which distributed the abrasive grain, and a lot of wash water and abrasive grains are needed. A lot of current, such wash water, and abrasive grains are disposed of as waste fluid, and these reuse is desired from reduction of the load to an environment, and a viewpoint of a deployment of a resource. Moreover, although the commercial abrasive grain which consists of SiO2 grade is usually used as an abrasive grain used for a CMP process, these are very expensive and to lessen the amount of the abrasive grain used also from an economical viewpoint is desired.
[0004]
[Problem(s) to be Solved by the Invention] As for wash water, an abrasive grain, etc. which are used for a CMP process as described above, it is desirable to reduce and reuse the amount of disposal from a deployment of a resource, consideration of an environment, and an economical viewpoint. Especially the abrasive grain used for a CMP process is very expensive, and it is desirable to decrease the amount used as much as possible.
[0005] However, since the slurry used once is diluted with wash water etc., slurry concentration falls and the pH value is also changing to the neutrality side. If slurry concentration falls, it may become impossible to grind a request. If the pH value of a slurry changes to a neutrality side, condensation of an abrasive grain occurs and it may become impossible moreover, to grind a request.
[0006] Furthermore, polish waste, for example, the piece of a pad, the piece of a metal, the piece of an oxide film, etc. are mixed in the slurry used once. When a CMP process is performed using a slurry which was described above, it becomes impossible to perform suitable polish. Precise processing is called for in processing of the semiconductor device with which recent years were integrated especially highly, and it is important to keep the property of a slurry constant also from this point.
[0007] This invention is made in order to solve a technical problem which was described above, and it aims at offering the slurry playback approach for obtaining the slurry which has a property equivalent to a new slurry, and the slurry regenerative apparatus using it while reducing the amount of the slurry to discard and raising the regeneration rate of a slurry.
[0008]

[Means for Solving the Problem] The slurry playback approach of this invention is characterized by to provide the slurry concentration process of removing a part of said dispersion medium from said slurry, pH adjustment process of adjusting said condensed pH value of a slurry, and the recovery process that collects the abrasive grains and the dispersion media below predetermined particle size from the slurry to which said pH value was adjusted in the playback approach of the slurry which has an abrasive grain, a dispersion medium, and impurities (polish waste, the piece of a pad, the piece of a metal, piece of an oxide film, etc.).

[0009] Moreover, the slurry playback approach of this invention is characterized by providing pH adjustment process of adjusting the pH value of said slurry, the slurry concentration process of removing said a part of dispersion medium from the slurry to which said pH value was adjusted, and the recovery process that collects the abrasive grains and dispersion media below predetermined particle size from said condensed slurry in the playback approach of the slurry which has an abrasive grain, a dispersion medium, and an impurity.

[0010] By the slurry playback approach of this invention, these may be performed to coincidence besides performing a slurry concentration process and pH adjustment process separately, as described above.

[0011] Said concentration process can contact said slurry in the filter for concentration which passes only a dispersion medium, for example, and can be performed by removing the dispersion medium which passed this filter for concentration.

[0012] As for said pH value, adjusting within the limits of specification is desirable, for example, it is within the limits of 10-11 in the slurry for oxide films. Adjustment of such a pH value can be performed by adding dispersants, such as a potassium hydroxide.

[0013] Said recovery process can be performed by collecting the components which the filter for recovery which passes the abrasive grain below a dispersion medium and predetermined particle size was made to pass said slurry for example, and passed this filter for recovery.

[0014] Moreover, the slurry regenerative apparatus of this invention is characterized by providing the circulation section which has the enriching section which removes a part of dispersion medium from said slurry, and pH controller which adjusts the pH value of said slurry, and the stripping section which is connected with said circulation section and collects the abrasive grains below a dispersion medium and predetermined particle size from said slurry in the regenerative apparatus of the slurry which has an abrasive grain, a dispersion medium, and an impurity.

[0015] Said enriching section possesses the filter for concentration which passes only a dispersion medium, and said slurry stripping section possesses the filter for recovery which passes the abrasive grain below a dispersion medium and predetermined particle size.

[0016]

[Embodiment of the Invention] The gestalt of operation of this invention is explained.

[0017] Drawing 1 shows the example of the slurry playback approach of this invention. The slurry playback approach shown in drawing 1 (a) removes first a dispersion medium (liquid component which consists of moisture, a dispersant, a penetrant remover, etc.) more unnecessary than a used slurry, condenses a slurry, adds a dispersant to this condensed slurry, adjusts pH, and collects the abrasive grains and dispersion media which are needed at the end. The slurry playback approach shown in drawing 1 (b) removes a dispersion medium more unnecessary than the slurry to which the dispersant was first added to the used slurry, pH was adjusted, and this pH was adjusted, condenses a slurry, and collects the abrasive grains and dispersion media which are needed at the end.

[0018] You may condense performing pH adjustment, as it is not necessary to necessarily perform such concentration and pH adjustment separately for example, and is shown in drawing 1 (c). Thus, efficient recovery of an abrasive grain is attained by adjusting pH before the process which collects the abrasive grains and dispersion media which are needed.

[0019] Hereafter, drawing 1 (a) is explained as an example about the slurry playback approach of this invention. Drawing 2 shows an example which showed the concentration approach in a concentration process. In addition, in drawing 2 , drawing right-hand side is a concentration side, and shows the slurry

condensed. Drawing left-hand side is a discharge side, and shows the dispersion medium removed from a slurry. In this invention, what it considers [ what ] as the filter 1 for concentration, for example, mainly passes only a dispersion medium 2, and passes neither an abrasive grain 3 nor an impurity 4 is used. As an abrasive grain 3, for example as an object for oxide films, cerium oxide (CeO2), a silica (SiO2), etc. are mentioned, and an alumina (aluminum 2O3) etc. is mentioned as W, aluminum, and an object for Cu. Although it changes as a filter for concentration also with abrasive grains to be used, the hollow fiber whose aperture is 0.01-0.15 micrometers, for example is mentioned. As an object for oxide films, an about 0.05-micrometer thing has a desirable aperture especially as about 0.1 micrometers and an object for W.

[0020] The slurry which you are going to make it condense from the concentration side of such a filter 1 for concentration, for example, a used slurry, is contacted, and the filter 1 for concentration is passed and a part of dispersion medium 2 is removed. Under the present circumstances, the abrasive grain 3 and impurity 4 which cannot pass the filter 1 for concentration remain to the slurry by the side of concentration. Since separation removal only of the dispersion medium can mainly be carried out according to this concentration process, the concentration of the slurry by the side of concentration can be raised. In addition, what is necessary is to remove no dispersion media and just to remove the dispersion medium of a superfluous part to the slurry concentration considered as a request at this concentration process.

[0021] Moreover, pH adjustment process in this invention adds a dispersant to a slurry, and adjusts the pH value of a slurry. Since the penetrant remover etc. is mixed in a used slurry, the pH value is changing to the neutrality side rather than the new slurry. Thus, when a pH value changes, an abrasive grain etc. will condense. In such a case, when the recovery in the recovery process explained later becomes low and the reuse of such a slurry is carried out with CMP equipment etc., it may differ from a slurry with the new homogeneity of polish etc. In this invention, a technical problem which was described above is cancelable by adjusting the pH value of a slurry.

[0022] As a dispersant for performing such pH adjustment, KOH (potassium hydroxide), NH4OH (ammonium hydroxide), etc. can perform an organic acid etc. to W and the slurry for aluminum by adding H2O2 (hydrogen peroxide) etc. at the slurry for oxide films at the slurry for Cu, for example. Within the limits of 10-11, by Cu, W, and the slurry for aluminum, the pH value of a slurry is considering as within the limits of 2-3, cancels condensation of an abrasive grain and becomes possible [ acquiring a polish property almost equivalent to a new slurry ] for example, at the slurry for oxide films.

[0023] Moreover, drawing 3 shows the recovery approach in a recovery process. Drawing left-hand side is a collected side, and the slurry to which the thing containing the component which should be collected, for example, the above-mentioned concentration, pH adjustment, etc. were performed is arranged. Moreover, drawing right-hand side is a recovery side, and the dispersion medium 2 and abrasive grain 3 which were collected from the collected side are located.

[0024] At the recovery process in this invention, as a filter 5 for recovery, the impurity of the magnitude more than fixed is not passed and the filter 5 for recovery which passes only the dispersion medium and abrasive grain which are considered as a request is used. Although it changes as such a filter for recovery also with abrasive grains to be used, the membrane filter whose aperture is 0.1-3 micrometers, for example is mentioned. Especially, in the case of the slurry for oxide films, that whose aperture is about 1 micrometer is desirable.

[0025] If the slurry by which concentration and pH adjustment were performed to the side collected such filters 5 for recovery is arranged, since the abrasive grain 3 and dispersion medium 2 which are contained in a used slurry can pass the filter 5 for recovery, they will move to a recovery side. Moreover, like an impurity 4, since a big thing cannot pass the filter 5 for recovery, it remains to a collected side.

[0026] Thus, the slurry in which an impurity is not contained can be obtained by collecting only what passed the filter 5 for recovery. Moreover, since a dispersion medium 2 and an abrasive grain 3 can be passed at an equivalent rate if such a filter 5 for recovery is used, it is not necessary to adjust concentration again by performing a recovery process, after adjusting the concentration of a collected

side appropriately. Especially, the pH value of a slurry is adjusted appropriately and it becomes recoverable [ an efficient abrasive grain ] by arranging to a collected side, after canceling condensation of an abrasive grain. Thus, the collected slurry can be again used with CMP equipment.
[0027] As explained above, while mainly removing a dispersion medium from a slurry in this invention and adjusting the concentration of a slurry, the component to discard is lessened as much as possible by collecting only required components and not collecting unnecessary components from a slurry, and it becomes possible to raise the regeneration efficiency of a slurry. Moreover, efficient recovery of a slurry also becomes possible by performing pH adjustment of a slurry before a recovery process.
[0028] Next, the slurry regenerative apparatus of this invention is explained.
[0029] Drawing 4 is the schematic diagram having shown an example of the slurry regenerative apparatus of this invention. A new slurry is sent to CMP equipment 7 from the slurry installation tubing 6. By being used with this CMP equipment 7, a penetrant remover, pad waste, etc. mix in a new slurry, and it becomes a used slurry. End storage of the used slurry is carried out through the used slurry exhaust pipe 8 to a storage tank 9. And it is sent to the circulation section 11 through piping 10 from a storage tank 9.
[0030] The circulation section 11 consists of piping 13 and 14 for circulation connected to a recycle tank 12 and this, and the enriching section 15, the density measurement section 16, and the pH controller 17 are arranged in the middle of this circuit.
[0031] The used slurry stored in the recycle tank 12 is sent to the enriching section 15 through the piping 13 for circulation. The filter 1 for concentration (dispersion-medium removal filter) which is made to pass only a dispersion medium as shown in drawing 1 , and is removed is formed in this enriching section 15. Among the slurries sent to the enriching section 15, a part of dispersion medium passes the filter 1 for concentration, and it is discharged through the dispersion-medium exhaust pipe 18 outside. Moreover, the abrasive grain, impurity, and dispersion medium which did not pass the filter 1 for concentration are returned to a recycle tank 12 through the piping 14 for circulation connected to the enriching section 15.
[0032] Thus, a superfluous dispersion medium can be removed from a slurry and the concentration of a slurry can be raised by circulating a slurry through the enriching section 15. It supervises by the density measurement section 16 prepared in the middle of the circuit, and the concentration of this slurry can close the valve 19 prepared in the piping 18 for dispersion-medium discharge when reaching predetermined concentration, can stop discharge of a dispersion medium, and can control the rise of concentration.
[0033] Next, circulating a slurry again, where a valve 19 is closed, from the pH controller 17, a dispersant is added and a pH value is adjusted to a predetermined value. By adjusting a pH value, condensation of an abrasive grain can be prevented and efficient recovery can be enabled at the following recovery process.
[0034] This pH adjustment may be performed before concentration, and you may carry out to concentration and coincidence. In performing concentration and pH adjustment to coincidence, while circulating a slurry where the valve 19 prepared in the piping 18 for dispersion-medium discharge is opened and removing a dispersion medium, from the pH controller 17, a dispersant is added and pH adjustment is performed. By doing in this way, pH adjustment and concentration can be performed to coincidence and the playback time amount of a slurry can be shortened sharply.
[0035] Next, the slurry to which concentration and pH adjustment were performed is sent to a stripping section 20. A stripping section 20 mainly consists of a tank 21 for recovery, and a filter 5 for recovery. It seems that the filter 5 for recovery does not pass an impurity and passes only a dispersion medium and an abrasive grain as shown in drawing 3 . A hollow fiber etc. can be used as such a filter.
[0036] It is made for this filter 5 for recovery to contact the slurry by which the side collected [ that ] was stored in the tank 21 for recovery, and the recovery side is connected to the piping 22 for recovery. Among the slurries stored in the tank 21 for recovery, the abrasive grain below a dispersion medium and fixed particle size passes the filter 5 for recovery, through the piping 22 for recovery, it is sent to CMP equipment 7 and a reuse is carried out. Moreover, the impurity which cannot pass the filter 5 for

recovery is accumulated in the tank 21 for recovery as it is.
[0037]
[Example] Hereafter, the gestalt of operation of this invention is explained with reference to an example.

[0038] The equipment of a configuration as shown in drawing 4 as a slurry regenerative apparatus was produced. It became CMP equipment mainly from SiO2 (particle size about 0.05-1 micrometer), the new slurry (made in the Cabot corporation, SC-1) which contains KOH (potassium hydroxide) as a dispersant was introduced, and the used slurry discharged from this CMP equipment was obtained. This used slurry was introduced into the circulation section, KOH which is a dispersant was added to concentration and coincidence, and pH adjustment was performed to them. In addition, the pH value of a slurry was adjusted to 11, using MS-8102 (0.1 micrometers of apertures) as a filter for concentration.
[0039] Next, the slurry to which concentration and pH adjustment were performed was introduced to the tank for recovery, the dispersion media and abrasive grains which are considered as a request from a slurry using the filter for recovery were collected, and it considered as the playback slurry. In addition, as a filter for recovery, CMP410-10 UV-M3 (1 micrometer of apertures) was used.
[0040] Thus, the particle size distribution of the obtained playback slurry are shown in drawing 5 . As shown in drawing 5 , there was no impurity near 3 micrometers - 8 micrometer which was looked at by the used slurry in the playback slurry obtained with the slurry regenerative apparatus of this invention, and particle size distribution also became almost equivalent to a new slurry.
[0041] Furthermore, the wafer was actually ground using such a playback slurry, and the polish rate (A/min) and the ground homogeneity (irregularity of a wafer) of a wafer were measured. A result is shown in drawing 6 and drawing 7 together with the case where a new slurry is used.
[0042] As shown in drawing 6 and drawing 7 , there is no great difference at the case where the case where a playback slurry is used also in which sample, and a new slurry are used, and it was admitted that the playback slurry of this invention had a property almost equivalent to a new slurry.
[0043]
[Effect of the Invention] According to the slurry playback approach of this invention, and the regenerative apparatus, it becomes possible from a used slurry to realize efficient playback of a used slurry by performing concentration and pH adjustment before the process which collects the abrasive grains below the particle size considered as a request.

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TECHNICAL FIELD

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[Field of the Invention] This invention reduces the amount of the slurry discarded especially about the slurry regenerative apparatus using the slurry playback approach and it which reproduce a used slurry, and relates to the slurry regenerative apparatus using the slurry playback approach and it which raised the regeneration rate of a slurry.

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PRIOR ART

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[Description of the Prior Art] In current and semiconductor production, CMP (Chemical Mechanical Polishing: chemical mechanical polish) serves as an indispensable technique. This CMP is in the inclination which it is applied also to metal wiring including an oxide film, and that applicability expands further.
[0003] At a CMP process, flattening processing of the device front face is carried out using the slurry which distributed the abrasive grain, and a lot of wash water and abrasive grains are needed. A lot of current, such wash water, and abrasive grains are disposed of as waste fluid, and these reuse is desired from reduction of the load to an environment, and a viewpoint of a deployment of a resource. Moreover, although the commercial abrasive grain which consists of SiO2 grade is usually used as an abrasive grain used for a CMP process, these are very expensive and to lessen the amount of the abrasive grain used also from an economical viewpoint is desired.

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## EFFECT OF THE INVENTION

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[Effect of the Invention] According to the slurry playback approach of this invention, and the regenerative apparatus, it becomes possible from a used slurry to realize efficient playback of a used slurry by performing concentration and pH adjustment before the process which collects the abrasive grains below the particle size considered as a request.

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TECHNICAL PROBLEM

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[Problem(s) to be Solved by the Invention] As for wash water, an abrasive grain, etc. which are used for a CMP process as described above, it is desirable to reduce and reuse the amount of disposal from a deployment of a resource, consideration of an environment, and an economical viewpoint. Especially the abrasive grain used for a CMP process is very expensive, and it is desirable to decrease the amount used as much as possible.
[0005] However, since the slurry used once is diluted with wash water etc., slurry concentration falls and the pH value is also changing to the neutrality side. If slurry concentration falls, it may become impossible to grind a request. If the pH value of a slurry changes to a neutrality side, condensation of an abrasive grain occurs and it may become impossible moreover, to grind a request.
[0006] Furthermore, polish waste, for example, the piece of a pad, the piece of a metal, the piece of an oxide film, etc. are mixed in the slurry used once. When a CMP process is performed using a slurry which was described above, it becomes impossible to perform suitable polish. Precise processing is called for in processing of the semiconductor device with which recent years were integrated especially highly, and it is important to keep the property of a slurry constant also from this point.
[0007] This invention is made in order to solve a technical problem which was described above, and it aims at offering the slurry playback approach for obtaining the slurry which has a property equivalent to a new slurry, and the slurry regenerative apparatus using it while reducing the amount of the slurry to discard and raising the regeneration rate of a slurry.

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MEANS

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[Means for Solving the Problem] The slurry playback approach of this invention is characterized by to provide the slurry concentration process of removing a part of said dispersion medium from said slurry, pH adjustment process of adjusting said condensed pH value of a slurry, and the recovery process that collects the abrasive grains and the dispersion media below predetermined particle size from the slurry to which said pH value was adjusted in the playback approach of the slurry which has an abrasive grain, a dispersion medium, and impurities (polish waste, the piece of a pad, the piece of a metal, piece of an oxide film, etc.).
[0009] Moreover, the slurry playback approach of this invention is characterized by providing pH adjustment process of adjusting the pH value of said slurry, the slurry concentration process of removing said a part of dispersion medium from the slurry to which said pH value was adjusted, and the recovery process that collects the abrasive grains and dispersion media below predetermined particle size from said condensed slurry in the playback approach of the slurry which has an abrasive grain, a dispersion medium, and an impurity.
[0010] By the slurry playback approach of this invention, these may be performed to coincidence besides performing a slurry concentration process and pH adjustment process separately, as described above.
[0011] Said concentration process can contact said slurry in the filter for concentration which passes only a dispersion medium, for example, and can be performed by removing the dispersion medium which passed this filter for concentration.
[0012] As for said pH value, adjusting within the limits of specification is desirable, for example, it is within the limits of 10-11 in the slurry for oxide films. Adjustment of such a pH value can be performed by adding dispersants, such as a potassium hydroxide.
[0013] Said recovery process can be performed by collecting the components which the filter for recovery which passes the abrasive grain below a dispersion medium and predetermined particle size was made to pass said slurry for example, and passed this filter for recovery.
[0014] Moreover, the slurry regenerative apparatus of this invention is characterized by providing the circulation section which has the enriching section which removes a part of dispersion medium from said slurry, and pH controller which adjusts the pH value of said slurry, and the stripping section which is connected with said circulation section and collects the abrasive grains below a dispersion medium and predetermined particle size from said slurry in the regenerative apparatus of the slurry which has an abrasive grain, a dispersion medium, and an impurity.
[0015] Said enriching section possesses the filter for concentration which passes only a dispersion medium, and said slurry stripping section possesses the filter for recovery which passes the abrasive grain below a dispersion medium and predetermined particle size.
[0016]
[Embodiment of the Invention] The gestalt of operation of this invention is explained.
[0017] Drawing 1 shows the example of the slurry playback approach of this invention. The slurry playback approach shown in drawing 1 (a) removes first a dispersion medium (liquid component which

consists of moisture, a dispersant, a penetrant remover, etc.) more unnecessary than a used slurry, condenses a slurry, adds a dispersant to this condensed slurry, adjusts pH, and collects the abrasive grains and dispersion media which are needed at the end. The slurry playback approach shown in drawing 1 (b) removes a dispersion medium more unnecessary than the slurry to which the dispersant was first added to the used slurry, pH was adjusted, and this pH was adjusted, condenses a slurry, and collects the abrasive grains and dispersion media which are needed at the end.

[0018] You may condense performing pH adjustment, as it is not necessary to necessarily perform such concentration and pH adjustment separately for example, and is shown in drawing 1 (c). Thus, efficient recovery of an abrasive grain is attained by adjusting pH before the process which collects the abrasive grains and dispersion media which are needed.

[0019] Hereafter, drawing 1 (a) is explained as an example about the slurry playback approach of this invention. Drawing 2 shows an example which showed the concentration approach in a concentration process. In addition, in drawing 2 , drawing right-hand side is a concentration side, and shows the slurry condensed. Drawing left-hand side is a discharge side, and shows the dispersion medium removed from a slurry. In this invention, what it considers [ what ] as the filter 1 for concentration, for example, mainly passes only a dispersion medium 2, and passes neither an abrasive grain 3 nor an impurity 4 is used. As an abrasive grain 3, for example as an object for oxide films, cerium oxide (CeO2), a silica (SiO2), etc. are mentioned, and an alumina (aluminum 2O3) etc. is mentioned as W, aluminum, and an object for Cu. Although it changes as a filter for concentration also with abrasive grains to be used, the hollow fiber whose aperture is 0.01-0.15 micrometers, for example is mentioned. As an object for oxide films, an about 0.05-micrometer thing has a desirable aperture especially as about 0.1 micrometers and an object for W.

[0020] The slurry which you are going to make it condense from the concentration side of such a filter 1 for concentration, for example, a used slurry, is contacted, and the filter 1 for concentration is passed and a part of dispersion medium 2 is removed. Under the present circumstances, the abrasive grain 3 and impurity 4 which cannot pass the filter 1 for concentration remain to the slurry by the side of concentration. Since separation removal only of the dispersion medium can mainly be carried out according to this concentration process, the concentration of the slurry by the side of concentration can be raised. In addition, what is necessary is to remove no dispersion media and just to remove the dispersion medium of a superfluous part to the slurry concentration considered as a request at this concentration process.

[0021] Moreover, pH adjustment process in this invention adds a dispersant to a slurry, and adjusts the pH value of a slurry. Since the penetrant remover etc. is mixed in a used slurry, the pH value is changing to the neutrality side rather than the new slurry. Thus, when a pH value changes, an abrasive grain etc. will condense. In such a case, when the recovery in the recovery process explained later becomes low and the reuse of such a slurry is carried out with CMP equipment etc., it may differ from a slurry with the new homogeneity of polish etc. In this invention, a technical problem which was described above is cancelable by adjusting the pH value of a slurry.

[0022] As a dispersant for performing such pH adjustment, KOH (potassium hydroxide), NH4OH (ammonium hydroxide), etc. can perform an organic acid etc. to W and the slurry for aluminum by adding H2O2 (hydrogen peroxide) etc. at the slurry for oxide films at the slurry for Cu, for example. Within the limits of 10-11, by Cu, W, and the slurry for aluminum, the pH value of a slurry is considering as within the limits of 2-3, cancels condensation of an abrasive grain and becomes possible [ acquiring a polish property almost equivalent to a new slurry ] for example, at the slurry for oxide films.

[0023] Moreover, drawing 3 shows the recovery approach in a recovery process. Drawing left-hand side is a collected side, and the slurry to which the thing containing the component which should be collected, for example, the above-mentioned concentration, pH adjustment, etc. were performed is arranged. Moreover, drawing right-hand side is a recovery side, and the dispersion medium 2 and abrasive grain 3 which were collected from the collected side are located.

[0024] At the recovery process in this invention, as a filter 5 for recovery, the impurity of the magnitude

more than fixed is not passed and the filter 5 for recovery which passes only the dispersion medium and abrasive grain which are considered as a request is used. Although it changes as such a filter for recovery also with abrasive grains to be used, the membrane filter whose aperture is 0.1-3 micrometers, for example is mentioned. Especially, in the case of the slurry for oxide films, that whose aperture is about 1 micrometer is desirable.

[0025] If the slurry by which concentration and pH adjustment were performed to the side collected such filters 5 for recovery is arranged, since the abrasive grain 3 and dispersion medium 2 which are contained in a used slurry can pass the filter 5 for recovery, they will move to a recovery side. Moreover, like an impurity 4, since a big thing cannot pass the filter 5 for recovery, it remains to a collected side.

[0026] Thus, the slurry in which an impurity is not contained can be obtained by collecting only what passed the filter 5 for recovery. Moreover, since a dispersion medium 2 and an abrasive grain 3 can be passed at an equivalent rate if such a filter 5 for recovery is used, it is not necessary to adjust concentration again by performing a recovery process, after adjusting the concentration of a collected side appropriately. Especially, the pH value of a slurry is adjusted appropriately and it becomes recoverable [ an efficient abrasive grain ] by arranging to a collected side, after canceling condensation of an abrasive grain. Thus, the collected slurry can be again used with CMP equipment.

[0027] As explained above, while mainly removing a dispersion medium from a slurry in this invention and adjusting the concentration of a slurry, the component to discard is lessened as much as possible by collecting only required components and not collecting unnecessary components from a slurry, and it becomes possible to raise the regeneration efficiency of a slurry. Moreover, efficient recovery of a slurry also becomes possible by performing pH adjustment of a slurry before a recovery process.

[0028] Next, the slurry regenerative apparatus of this invention is explained.

[0029] Drawing 4 is the schematic diagram having shown an example of the slurry regenerative apparatus of this invention. A new slurry is sent to CMP equipment 7 from the slurry installation tubing 6. By being used with this CMP equipment 7, a penetrant remover, pad waste, etc. mix in a new slurry, and it becomes a used slurry. End storage of the used slurry is carried out through the used slurry exhaust pipe 8 to a storage tank 9. And it is sent to the circulation section 11 through piping 10 from a storage tank 9.

[0030] The circulation section 11 consists of piping 13 and 14 for circulation connected to a recycle tank 12 and this, and the enriching section 15, the density measurement section 16, and the pH controller 17 are arranged in the middle of this circuit.

[0031] The used slurry stored in the recycle tank 12 is sent to the enriching section 15 through the piping 13 for circulation. The filter 1 for concentration (dispersion-medium removal filter) which is made to pass only a dispersion medium as shown in drawing 1 , and is removed is formed in this enriching section 15. Among the slurries sent to the enriching section 15, a part of dispersion medium passes the filter 1 for concentration, and it is discharged through the dispersion-medium exhaust pipe 18 outside. Moreover, the abrasive grain, impurity, and dispersion medium which did not pass the filter 1 for concentration are returned to a recycle tank 12 through the piping 14 for circulation connected to the enriching section 15.

[0032] Thus, a superfluous dispersion medium can be removed from a slurry and the concentration of a slurry can be raised by circulating a slurry through the enriching section 15. It supervises by the density measurement section 16 prepared in the middle of the circuit, and the concentration of this slurry can close the valve 19 prepared in the piping 18 for dispersion-medium discharge when reaching predetermined concentration, can stop discharge of a dispersion medium, and can control the rise of concentration.

[0033] Next, circulating a slurry again, where a valve 19 is closed, from the pH controller 17, a dispersant is added and a pH value is adjusted to a predetermined value. By adjusting a pH value, condensation of an abrasive grain can be prevented and efficient recovery can be enabled at the following recovery process.

[0034] This pH adjustment may be performed before concentration, and you may carry out to concentration and coincidence. In performing concentration and pH adjustment to coincidence, while

circulating a slurry where the valve 19 prepared in the piping 18 for dispersion-medium discharge is opened and removing a dispersion medium, from the pH controller 17, a dispersant is added and pH adjustment is performed. By doing in this way, pH adjustment and concentration can be performed to coincidence and the playback time amount of a slurry can be shortened sharply.

[0035] Next, the slurry to which concentration and pH adjustment were performed is sent to a stripping section 20. A stripping section 20 mainly consists of a tank 21 for recovery, and a filter 5 for recovery. It seems that the filter 5 for recovery does not pass an impurity and passes only a dispersion medium and an abrasive grain as shown in drawing 3 . A hollow fiber etc. can be used as such a filter.

[0036] It is made for this filter 5 for recovery to contact the slurry by which the side collected [ that ] was stored in the tank 21 for recovery, and the recovery side is connected to the piping 22 for recovery. Among the slurries stored in the tank 21 for recovery, the abrasive grain below a dispersion medium and fixed particle size passes the filter 5 for recovery, through the piping 22 for recovery, it is sent to CMP equipment 7 and a reuse is carried out. Moreover, the impurity which cannot pass the filter 5 for recovery is accumulated in the tank 21 for recovery as it is.

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EXAMPLE

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[Example] Hereafter, the gestalt of operation of this invention is explained with reference to an example.

[0038] The equipment of a configuration as shown in drawing 4 as a slurry regenerative apparatus was produced. It became CMP equipment mainly from SiO2 (particle size about 0.05-1 micrometer), the new slurry (made in the Cabot corporation, SC-1) which contains KOH (potassium hydroxide) as a dispersant was introduced, and the used slurry discharged from this CMP equipment was obtained. This used slurry was introduced into the circulation section, KOH which is a dispersant was added to concentration and coincidence, and pH adjustment was performed to them. In addition, the pH value of a slurry was adjusted to 11, using MS-8102 (0.1 micrometers of apertures) as a filter for concentration.
[0039] Next, the slurry to which concentration and pH adjustment were performed was introduced to the tank for recovery, the dispersion media and abrasive grains which are considered as a request from a slurry using the filter for recovery were collected, and it considered as the playback slurry. In addition, as a filter for recovery, CMP410-10 UV-M3 (1 micrometer of apertures) was used.
[0040] Thus, the particle size distribution of the obtained playback slurry are shown in drawing 5 . As shown in drawing 5 , there was no impurity near 3 micrometers - 8 micrometer which was looked at by the used slurry in the playback slurry obtained with the slurry regenerative apparatus of this invention, and particle size distribution also became almost equivalent to a new slurry.
[0041] Furthermore, the wafer was actually ground using such a playback slurry, and the polish rate (A/min) and the ground homogeneity (irregularity of a wafer) of a wafer were measured. A result is shown in drawing 6 and drawing 7 together with the case where a new slurry is used.
[0042] As shown in drawing 6 and drawing 7 , there is no great difference at the case where the case where a playback slurry is used also in which sample, and a new slurry are used, and it was admitted that the playback slurry of this invention had a property almost equivalent to a new slurry.

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## DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]
[Drawing 1] Drawing having shown the process of the slurry playback approach of this invention.
[Drawing 2] The general-view Fig. having shown an example of the concentration approach in this invention.
[Drawing 3] The general-view Fig. having shown an example of the recovery approach in this invention.

[Drawing 4] The general-view Fig. having shown the slurry regenerative apparatus of this invention.
[Drawing 5] Drawing having shown the particle size distribution of a playback slurry.
[Drawing 6] Drawing having shown the result of the polish rate in the polish which used the playback slurry.
[Drawing 7] Drawing having shown the homogeneity of the wafer in the polish which used the playback slurry.
[Description of Notations]
1 .... Filter for concentration
5 .... Filter for recovery
11 .... Circulation section
15 .... Enriching section
17 .... pH controller
20 .... Stripping section

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[Translation done.]

* NOTICES *



1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

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DRAWINGS

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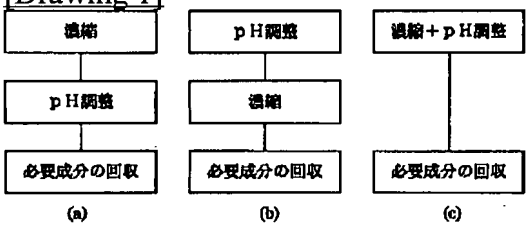
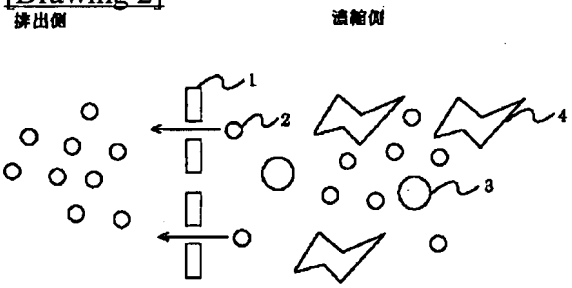
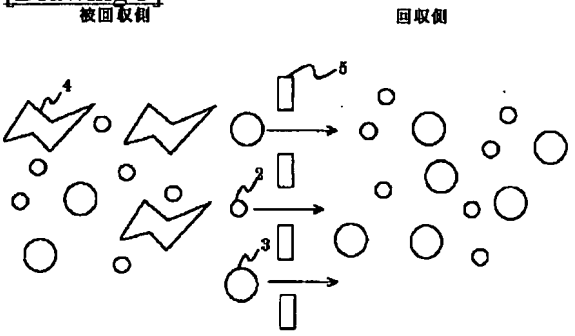
[Drawing 1]



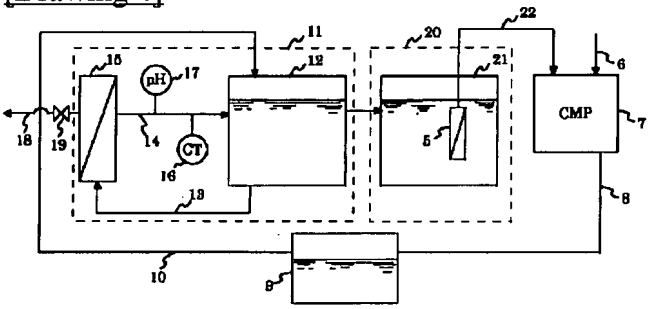

[Drawing 2]



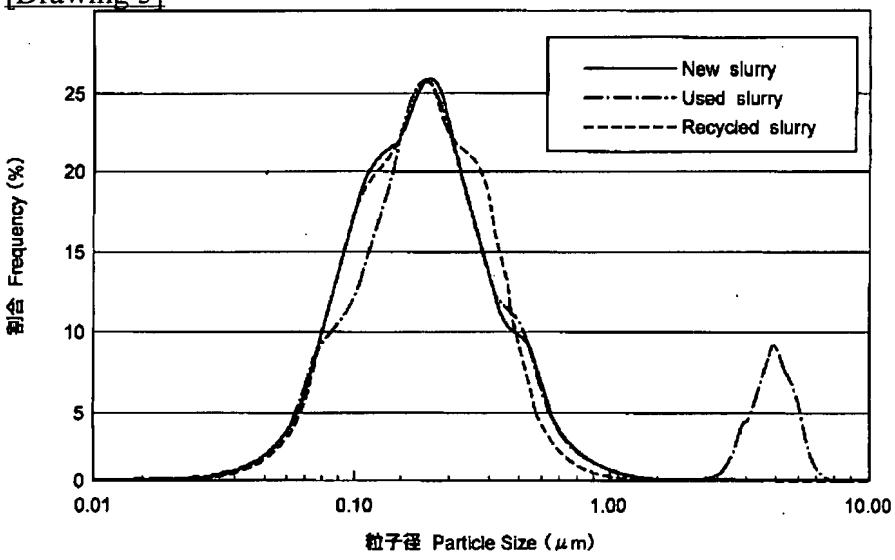

[Drawing 3]



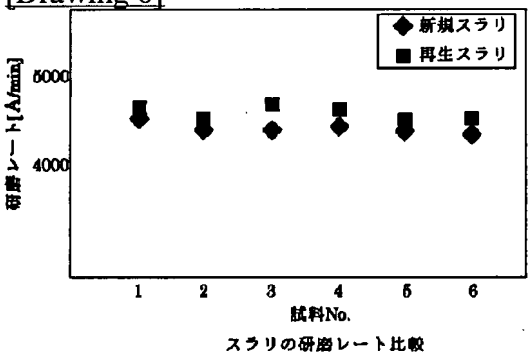

[Drawing 4]



[Drawing 5]

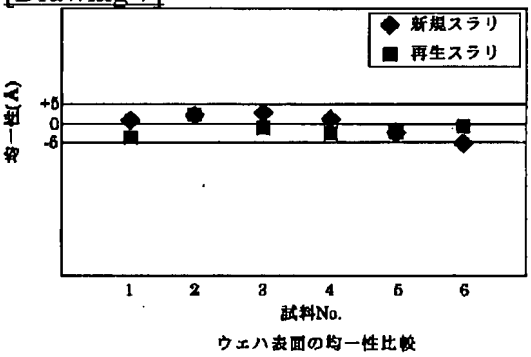



[Drawing 6]




スラリの研磨レート比較

[Drawing 7]




ウェハ表面の均一性比較

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[Translation done.]